

WM'02 Conference, February 24-28, 2002, Tucson, AZ

DEVELOPMENT AND DEPLOYMENT OF ADVANCED CORROSION MONITORING SYSTEMS FOR HIGH-LEVEL WASTE TANKS

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ABSTRACT

This paper describes the results of a collaborative technology development program, sponsored by the Tanks Focus Area, to use electrochemical noise (EN) for corrosion monitoring in underground storage tanks. These tanks, made of carbon or stainless steels, contain high-level radioactive liquid waste (HLW) generated by weapons production or radioactive liquid waste from nuclear fuel reprocessing activities at several Department of Energy (DOE) sites. The term EN is used to describe low frequency fluctuations in current and voltage measurements associated with corrosion. In their most basic form, EN-based corrosion monitoring systems measure and record these fluctuations over time from electrodes immersed in the environment of interest – in this case, radioactive tank waste. The resulting EN signals have characteristic patterns for different corrosion mechanisms.

In recent years, engineers and scientists from several DOE sites, in collaboration with several private companies, have conducted laboratory studies and field applications to correlate the EN signals with corrosion mechanisms active in the radioactive waste tanks. The participating DOE sites are Hanford, Savannah River, Oak Ridge Reservation and the Idaho National Engineering and Environmental Laboratory. The commercial vendors have included HiLine Engineering and Fabrication, Inc., EIC Laboratories, Inc., and AEA Technologies. Successful deployment of the EN technology will yield improved information of waste tank corrosion conditions, better tank management, and lower overall cost.

INTRODUCTION

Background

Underground storage tanks made of carbon or stainless steel, are used to contain radioactive liquid waste generated from weapons production and nuclear fuel reprocessing activities at several Department of Energy (DOE) sites. Corrosion and tank integrity are major issues concerning worker and environmental safety. Historically, corrosion monitoring and control have been provided through waste chemistry sampling and analysis programs or by monitoring of corrosion coupons placed in the tanks. In the first process, tank waste is sampled, analyzed and compared to guidelines established from an extensive laboratory test program, which used simulated waste. Therefore, tank wall corrosion is inferred from waste chemistry analysis. The corrosion coupon measurement method requires evaluation of coupons that simulate the materials and stress state of the tank materials and



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fabrication. Both the sampling and coupon exposure methods are expensive and time-consuming, with neither providing continuous real-time corrosion information.

In addition to coupon exposure, corrosion can be monitored through a variety of electrochemical techniques. A small number of these techniques have been tried within the DOE complex to determine the corrosivity of nuclear waste underground storage tanks (1). Coupon exposure programs, linear polarization resistance (LPR), and electrical resistance techniques have all been tried with limited degrees of success. These techniques have proven effective for monitoring uniform corrosion, but are not well suited for early detection of localized forms of corrosion such as pitting and stress corrosion cracking (SCC). Pitting and SCC have been identified as the primary modes of corrosion for the majority of the materials (carbon and stainless steel) used in the radioactive waste tanks (2-3).

In recent years, a new corrosion monitoring system has shown promise in detecting localized corrosion and measuring uniform corrosion rates in process industries (4-20). The system measures electrochemical noise (EN) generated by corrosion. The term EN is used to describe low frequency fluctuations in measured current and voltage. In their most basic form, EN-based corrosion monitoring systems track these fluctuations over time from three electrodes immersed in the environment of interest. The three electrodes, which are made of the same material, are called working, counter, and reference electrodes. Laboratory studies and field applications have shown that different types of corrosion create different patterns of current and voltage fluctuations. By monitoring the EN produced by corrosion on electrodes immersed in waste tanks, conditions for waste tank corrosion can be observed in real-time.

In late 1995, the DOE's Tanks Focus Area (TFA) launched an effort to develop EN technology for use in a wide variety of tanks and with disparate chemistries. The initial deployment of this promising technology used fixed-height, mast-mounted, systems for use by Hanford's corrosion control program in carbon steel tanks containing highly caustic waste. At the Savannah River Site (SRS), the EN technology was combined with Raman spectroscopy in an adjustable height probe. The Oak Ridge Reservation (ORR) added EN capability to one of their stainless steel tanks containing caustic waste with a system similar to those deployed at Hanford. Most recently, a program was initiated at the Idaho National Engineering and Environmental Laboratory (INEEL) to ascertain the applicability of EN-based systems to stainless steel tanks containing acidic waste solutions with high concentrations of chlorides and fluorides. This paper presents, for each of the DOE sites, a description of the EN development and probe designs, the current status of the corrosion monitoring systems at the sites, and the planned activities for future EN development.

EN Technology

Aqueous corrosion is an electrochemical process that leads to the degradation of metallic surfaces. The corrosion process proceeds by the formation of anodic and cathodic areas on the metallic surface. At the anodes, the metal corrodes by an oxidation reaction with the solution constituents, while at cathodic areas a reduction reaction occurs, such as oxygen reduction. During general corrosion these cathodic and anodic areas are continuously changing. The overall surface potential and the localized currents are, therefore, also changing or fluctuating. EN-based corrosion monitoring systems track these fluctuations in current and voltage over time from electrodes immersed in an environment of interest.

Three electrodes are needed to make the EN measurements. Two electrodes are coupled to monitor the current that flows between them. The electrodes are typically made of identical material, so that the corrosion process and resulting potentials will be similar. However, the small potential differences lead to a small current flow (μA -mA) that continuously fluctuates. The third electrode acts as the reference (often referred to as pseudo-reference) for the potential measurements of the coupled electrodes. This electrode is generally of identical material as the coupled electrodes so that potential measurements (mV) are also small.

Different patterns of current and voltage indicate different forms of active corrosion. Uniform corrosion EN data are characterized by small random fluctuations of current and potential as discussed above. For pitting corrosion the EN depends on the aggressiveness of the measured system. Singular pitting events in a system of very low aggressiveness produce well-defined singular transients in the current and potential EN data. As the number of

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active pits on an electrode increases (increasing solution aggressiveness), the resolution of these individual transients becomes more difficult and the EN data can approach the appearance of active uniform corrosion. Pit initiation events produce different EN data than pit growth events. Pit initiation transients rise quickly from a baseline EN fluctuation level and tend to have sharp points with rapid exponential decays back to the prior level of current and potential. Pit growth also produces a sharp movement in current and potential, but this movement is followed by a stable period of time at the newly attained level and then an exponential decay back to some baseline level seen prior to the pitting event.

During SCC, large, simultaneous, sharp movements in potential and current followed by exponential decay back to a baseline level characterize crack propagation EN data. Film rupture and crack advance followed by anodic dissolution, repassivation, and other catalyzed reactions produce the transients in the EN data – typically larger than transients associated with incipient pitting. The direction of a transient depends on the reactions that take place on the new crack surface. SCC transients decrease in size and frequency as stress is reduced.

Crevice corrosion is manifested in EN data as a series of cyclic, regularly spaced spikes in current and potential that start and stop as the crevice becomes active and inactive. Different systems produce different shaped transients in different directions based on the reactions occurring in the mouth of the crevice.

Many techniques exist for EN data analysis. Techniques range from traditional statistical analysis methods, visual inspection of raw data files, conversion of time domain data to frequency domain, and numerous other variations of these basic forms. In practice, no single analysis technique is likely to be perfect for every system. Currently, manual inspection of raw EN data is the primary mode of data analysis used within the DOE complex.

HANFORD SITE

Overview

In October 1995, initial proof-of-principle laboratory work produced the technical basis for using EN-based systems to monitor corrosion in Hanford's nuclear waste tanks (21). Program development was a joint venture including the TFA, various Hanford tanks operating contractors, and HiLine Engineering and Fabrication, Inc (HiLine). Waste storage tanks at Hanford are constructed of carbon steel and the wastes inside them are highly caustic. During this effort, a prototype system was constructed and deployed in double shell tank 241-AZ-101 in August 1996 (22). After the successful demonstration of this prototype, a larger full-scale system with more features was designed and installed into tank 241-AN-107 in September 1997 (23). In August of 1998 the second full-scale system, similar to the 241-AN-107 system, was designed, fabricated and installed in tank 241-AN-102 (24). A third system was installed in tank 241-AN-105 in January 2000 (25). In January 2001 a fourth system was installed in tank 241-AN-104 (26). In August 2001 a fifth probe, similar in design to the fourth system, was installed in tank 241-AN-107 to replace the original system installed in 1997.

Since the initial installation in 1996, each successive design evolved to correct previous problems. Additionally, each successive system has made better use of tank riser space by incorporating other equipment and monitoring techniques on the probe body to compliment the existing corrosion monitoring components.

By the time the tank 241-AN-104 system was installed in January 2001, many of the technical issues identified on previous systems had been resolved or mitigated. A decision was made to upgrade the aboveground electronics of several of the other systems. These upgrades were accomplished during the balance of fiscal year 2001. Additionally, all data collection computers were reconfigured and housed in a single location.

Tank 241-AZ-101 Prototype System Description

Several design goals were established for the initial system. They included a minimum two-year system service life; remote data acquisition and system control capability; minimal liquid retention of in-tank equipment for optimum

decontamination; minimal sensitivity to temperatures up to 100°C, pH ranges from 7 to 14, radiation levels up to 1000 R/hr, and liquid phase flow rates up to 1.0 m/s.

The prototype corrosion probe was constructed from 3.8-cm diameter AISI 304L stainless steel (UNS 30403) pipe for extended service in the waste tank environment. The probe was sized for a nominal 10.2-cm diameter tank riser and was approximately 10 meters long. It included three electrode arrays; two arrays were positioned to monitor vapor space corrosion, and the third array was below the waste surface. Each electrode was a C-ring (ASTM G-38) electrode and constructed of archived ASTM A537-Class 1 tank steel. The working electrode located in the waste was pre-cracked and strained for detection of SCC. The other two electrodes in the array were not strained or pre-cracked. The vapor space arrays were not pre-cracked, because pitting was the primary corrosion mechanism of concern.

A shielded data cable extended through the interior length of the sealed pipe. For each electrode, a wire was separated from the shielding and attached to the electrode. A commercially available glass to metal seal was used to penetrate the probe tree pipe wall while maintaining electrical isolation between the electrodes and the pipe wall. A gasket suitable for radiation environments isolated the electrodes from the glass-to-metal seals. The shielded cable terminated at the top to the probe tree assembly in a weather-tight box. An above ground cable ran from the top of the probe to the corrosion monitoring instrumentation.

The corrosion monitoring instrumentation was a commercially available electrochemical measurement system with an eight channel multiplexer. Galvanic corrosion operating software for this system was customized and optimized for EN data collection. Data were collected sequentially on the electrode arrays using three channels of the multiplexer. Potential and current measurements were recorded once per second on the active array. Data were typically collected for 600 seconds per channel. Post-collection data analysis was performed using internally developed statistical analysis software and a commercial spreadsheet. Data acquisition was terminated on the vapor space channels after nine months, as the EN system electronics did not have the necessary level of potential noise sensitivity to record meaningful information from these electrodes. All remaining components were removed from the tank in fiscal year 2000 for evaluation.

Although a great deal of data were collected from the single channel immersed in the waste, the instrument sensitivity was deemed to be too low and monitoring was discontinued in 1999. Electrochemical *current* noise data collected from this tank were of high quality, but the corresponding voltage noise data was not acceptable.

Tank 241-AN-107 System Description

Two requirements were changed from the AZ-101 prototype design for the original tank 241-AN-107 corrosion monitoring system. The required years of service were extended to five years instead of two, and the remote data acquisition and system control were through the local area network.

The AN-107 probe was constructed of two materials, 2.5-cm diameter schedule XXS AISI 304L pipe and 9.5-cm diameter AISI 304L pipe. The 9.5-cm pipe was filled with a low density concrete to serve as a shield plug for the riser. The probe was approximately 16 m in length. Along this length were four two-channel electrode arrays. The arrays were placed for monitoring in both the vapor space and waste. Of the total eight arrays four had C-rings (ASTM G-38) and the other four had bullet-shaped pin type electrodes (archived ASTM A537 carbon steel). To assist in monitoring SCC, one electrode of each C-ring array was pre-cracked and strained prior to immersion in the waste. The arrangement of the electrodes is shown in Figure 1.



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A 25-conductor full-shielded data cable extends through the length of the interior of the sealed pipe. The connections were the same as described above for the tank 241-AZ-101 probe. The aboveground cabling utilizes a grounded shield to protect the signal from external electrical interference and shunt-diode type intrinsic safety barriers to meet site safety requirements for waste intrusive instrumentation. All intrinsic safety barriers, drain wires, and the overall cable shielding are grounded to the tank riser at one location. The corrosion monitoring instrumentation was originally a DENIS eight-channel corrosion monitoring system manufactured by Capcis March Limited (CML). DENIS is a registered trademark of Capcis March Ltd., Manchester, UK.

Lack of funding restricted the data analysis to only that necessary to validate system performance. In 1998, data from the 241-AN-107 system were finally analyzed. The data quality was low due to external electrostatic noise picked up along the long data cable that leads from the in-tank probe to the data collection hardware housed outside the tank farm. In fiscal year 2001, the probe, all aboveground hardware, software, cabinetry, and cabling was removed and the system was reconfigured and reinstalled to match the Tank 241-AN-104 system described below.

Tank 241-AN-102 System Description

The features of the Tank 241-AN-102 corrosion monitoring system were similar to those for the Tank 241-AN-107 system, except for several significant changes. Improvements were made to the probe body, the data acquisition software, and the signal processing instrumentation. The electrical feed-throughs to which the electrodes were connected were angled to withstand probe flexure. Previously, a feed-through was perpendicular to the probe body, which also made fabrication difficult. The probe was equipped with eight thermocouples and a waste high-level detector. The probe body was grounded to shield against unwanted interference in the data.

New software and instrumentation were installed which, like the Tank 241-AN-107 system, simultaneously monitored eight channels of EN electrodes. The new system was automated and could be configured by the user. The operating software was also compatible with site standard desktop computers. The system was now housed in a climate controlled building outside the tank farm. The remaining features were similar to previous probes including a collection rate of one measurement per second, periodic performance of linear polar resistance scans, use of intrinsic safety barriers for all conductors and eight electrodes array (four bullet type and four C-ring). Further improvements were made to the data acquisition and transmission hardware after the initial installation.

Tank 241-AN-102 data revealed problems similar to those experienced with the 241-AN-107 system. Although the new system software greatly accelerated the data analysis process, external noise and interference in the data rendered the 241-AN-102 data questionable for use. These problems were associated with the data cable that utilized bundles of unshielded twisted pair conductors. When replaced with shielded twisted pairs, the problems associated with noise were eliminated.

The upgraded system matches the Tank 241-AN-105 system described below.

Tank 241-AN-105 System Description

The Tank 241-AN-105 corrosion monitoring system is a multi-function instrument and contains several features not seen on previous Hanford systems. In addition to the eight channels of corrosion monitoring electrodes incorporated on previous probes, the new system is also fitted with an array of 22 thermocouples, an adjustable height verification thermocouple, a tank waste high-level detector, ports for pressure/gas sampling and a set of strain gauges to monitor the effects of tank operations on the in-tank instrumentation. These features add a great deal of functionality to the probe, provide for a better understanding of the relationship between corrosion and other tank operating parameters, and optimize the use of the riser that houses the probe in the tank.

Other important improvements to the systems included passing both the structural analysis of mixer pump operation evaluation and the standard seismic analysis for non-safety class equipment, during the design process. An adjustable collar was added to the top of the probe so that the height could be changed during installation. A new design was used for the attachment of the electrodes to the probe body, which minimizes the exposure of the O-ring

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gasket to the waste (See Figure 2). Above ground wiring was fitted with driven shields to reduce external electrostatic noise in the data, and the system was linked to local area network to facilitate remote access.

The data collection software is an Amulet system manufactured by Corrosion & Condition Control, Ltd. The data collection hardware is a CIS400 instrument from Petroleum Research and Production, Ltd. Data quality from this system has been of high quality.

Tank 241-AN-104 System Description

The Tank 241-AN-104 corrosion monitoring system is identical to the 241-AN-105 system with the exception of the analog data cable between the probe top and instrumentation and the addition of a water lance to facilitate installation. On the Tank 241-AN-104 system, eight separate lengths (one per channel) of data cable are used to transmit signals from probe electrodes to the data collection hardware. This cable was selected to provide additional physical separation between conductors.

Independent System Review

In May 2001, the EN-based corrosion monitoring program was reviewed by a panel of experts from academia and industry to provide a path forward for the site for corrosion monitoring and integrity management of the tanks. The corrosion monitoring program was reviewed in terms of readiness for transition to operations, technical issues, and site integrity management issues. The panel issued a report in June 2001 that was generally favorable, but suggested areas for improvement before EN-based corrosion monitoring can be incorporated in day-to-day operations. Many of the technical findings matched those previously identified during an internal review. (27).

Specific recommendations from the external and internal reviews were as follows: (1) Develop and implement a long-term integrated tank integrity management plan which incorporates corrosion monitoring, non-destructive testing, and tank waste sampling/analysis; (2) Provide continuous funding for this program commensurate with long-term integrated program needs; (3) Conduct further laboratory work to develop or refine EN signal recognition and data reduction methodologies, to evaluate SCC detection and characterization, and to characterize gasket failure; (4) Conduct full-scale mockup testing of systems in available cold tank and/or well facilities; and (5) Conduct forensic analysis and investigations on probes removed from service.

SAVANNAH RIVER SITE

Overview

Real-time corrosion and chemistry data are necessary at SRS during waste processing since the tank and waste conditions are dynamic. Starting in 1997, the SRS began developing a remote, real-time monitoring probe for measuring the concentration of waste constituents which impact the tank corrosion and, in 1998 added the capability for detecting corrosion in high level waste tanks. The SRS probe incorporates two technologies: laser Raman spectroscopy (RS) for chemical analysis, and electrochemical noise (EN) for corrosion monitoring. Integration of these two technologies into one probe has the advantage of correlating real-time information of waste inhibitor concentrations and corrosion in an actual radioactive tank waste environment. Additionally, a deployable, variable-height sensor, such as this probe, provides the capability of waste characterization and corrosion monitoring as a function of depth and a more complete characterization of conditions. Probe development for SRS is a joint project of the Department of Energy Tanks Focus Area, Westinghouse Savannah River Company, the Savannah River Technology Center, EIC Laboratories, Inc. (EIC), and HiLine.

Waste tank corrosion monitoring and control at SRS is currently provided by process knowledge, extensive laboratory test results (which were obtained from simulated wastes) and chemical analyses of tank grab samples. The procedure for pulling a tank grab sample involves numerous steps, puts personnel at risk to exposure, and takes up to one month for results. The limitations of the current system in providing accurate information of the tank corrosion and waste conditions ultimately limits the assessments of tank structural integrity and overall safety. The

benefit of the SRS probe for real-time information of changing tank and waste conditions will reduce the need and cost associated with manual sampling, reduce the risk of personnel exposure, and improve the analysis of tank structural integrity and safety.

Monitoring Technologies

The laser RS technology was optimized to measure the primary waste constituents involved in corrosion of the SRS high-level waste tanks, specifically nitrate, nitrite, and hydroxide. The detection ranges for these anions are 0.02-3.2 M for nitrate, 0.05-3.0 M for nitrite, and 0.03-13.4 M for hydroxide. Other polyatomic molecules, such as sulfates, carbonates and chromates, could also be detected, but their detection was not optimized for the SRS probe. Raman analyses of waste tank anions in actual SRS tank waste have been conducted previously (28). The results indicated seven polyatomic anion, including nitrate, nitrite, and hydroxide, were readily analyzed with the RS technique. Another important result was that fluorescence was found not to interfere in obtaining quantitative data for anion concentration.

RS is an optical technique that produces a unique spectrum for every molecule, which results from inelastic scattered light collected for each molecule. In general, the spectral features for each molecule are sharp. Multiple components of mixtures can often be differentiated without physically separating them because of these "fingerprint" spectra. Although a full Raman instrument is unsuitable for in-tank deployment, small, flexible fiber optic probes connected to the instrument can be readily delivered into the tanks for *in situ* monitoring.

The EN technology, which is similar to that described previously for the Hanford probes, measures over time the instantaneous current and potential difference fluctuations that occur between three identical electrodes. These low frequency signals are from the interactions of the electrodes with the exposure solution, including corrosion. For SRS, the primary corrosion mechanisms of concern are general, pitting and SCC. Generally, the noise signals have a characteristic pattern for different corrosion processes. Previous laboratory work performed in SRS simulated waste solutions showed that EN was feasible for use in high-level waste tank chemistries (29). Prior to choosing the corrosion monitoring technique, other techniques were evaluated, but did not provide the flexibility and simplicity of EN.

SRS Probe Configuration

The SRS probe was required to be a height-adjustable measurement system because the primary use of the probe is for active tanks in which the waste composition is changing due to frequent additions and removals. The system was designed as a moveable probe head on a flexible cable. The cable is deployed from a reel mechanism housed in a confinement chamber on top of the tank riser to protect against radioactive contamination on the tank top. The cable connects to the reel hub from which the measurement cables run to the instrument trailer.

A multi-layered flexible cable is attached to the reel and houses the electrical, fiber optic and pneumatic cables and lines. The cable consists of a 2.54 cm (1-inch ID) corrugated stainless steel conduit for water tightness with an exterior stainless steel sheathing for structural support. The stainless steel conduit was chosen to resist the chemical environment of the waste. A polyolefin coating is the outer layer and provides an additional barrier to waste intrusion. The flexible cable houses the various leads for both the EN and RS techniques. There are two electrical cables for EN, and two fiber optic cables and a pneumatic tube for RS. Each electric cable contains two shielded twisted pairs of 22 gauge electric wire. One cable is for each electrode set. The fiber optic cables are for the excitation laser and signal collection. The pneumatic tube provides a way to introduce calibrating solutions, cleaning water, vacuum, and pressure to the RS sample chamber.

The flexible cable attaches to the top of the probe head with a swivel swag lock fitting. An early schematic of the probe body, which shows the internal layout of the probe head, is shown in Figure 2. The electrical wires separate from a cable and go to one of the electrodes. The wire for each electrode is soldered to the back of electrical feed through for the electrode. One of the electrical cables is for each probe set. The feed through, which was designed by Hi-Line, is placed into the sidewall of the probe head and secured with an O-ring and a collet ring. The bullet-shaped electrode screws on the external post of the feed-through with another O-ring that isolates the electrode from

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the probe body. This design allows for ease of assembly without jeopardizing the leak tightness of the probe head. The final design differed slightly from the preliminary schematic shown.

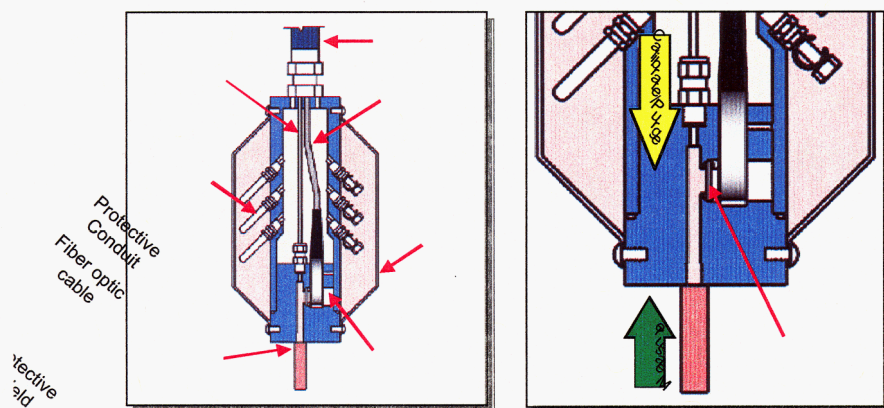


Fig. 2. Savannah River combined probe head schematic.

The electrodes are made from materials similar to the materials of construction for the waste tank wall and cooling coils. Archived materials from the actual construction were not available so the electrodes were fabricated from new stock material. One set was made from A537 carbon steel representing the tank wall; the other set was made from A106 carbon steel representing the cooling coils. The surfaces of the electrodes have a 600-grit finish so they will have a minimal oxide layer. Since these materials are in a pristine condition, they do not replicate the tank wall surface. Depending on the location, the tank wall has a build up of oxide, corrosion products, salt residues, and sludge resulting from the exposure to various waste streams for more than twenty years. However, the electrodes are expected to provide a conservative response since the metal surface is directly exposed to the waste.

The probe head is constructed of two stainless steel pieces, which have a screw connection. The upper pipe section supports the EN electrodes and houses the various wires and tubes. After assembly of the probe head the upper section was filled with a silicone rubber potting material. The RS probe and sample chamber are contained in the lower section along with the thermocouple. The RS laser probe fits snugly into the lower section. The laser window is oriented toward the sample chamber separated by a sapphire window. The polyethylene tube is connected to the top of the sample chamber. The tube provides a path for the calibrating solution, air to remove chamber contents, and vacuum to fill the chamber with waste. The bottom of the chamber has a filter for removing solids from the waste sample.

Monitoring Instrumentation

The data acquisition instrumentation for EN consists of an intrinsic safety barrier (ISB), voltage and current measuring unit (corrosion rack), and a control computer for data storage and analysis. The layout for the system is such that the analog voltage and current signals from the electrodes pass through an ISB to the corrosion rack. A computer that contains the data acquisition software is connected to the corrosion rack by an RS232 cable. Up to 40 megabytes of data can be temporarily stored in the corrosion rack prior to transfer to the computer. The ISB protects the wiring and electrodes from any fault that occurs in the instrumentation or line voltage. Because the interior of the tank contains some hydrogen (although below the flammability limit) the ISB provides a preventive safety measure. The ISB contains four 2-channel shunt diode safety barriers; two shunt diodes for each set of electrodes. The three electrode leads and the grounding wire for the cable pass through the ISB. The electrodes are connected to the grounding bar in the ISB that is connected to the current unit ground. Thus, grounding for all parts of the system are to the same location.

The RS instrumentation consists of an echelle spectrograph, laser, and pumps. An EIC echelle spectrograph with a thermoelectrically cooled charge coupled device (CCD) detector will be used for Raman spectral acquisition. The data acquisition software (RS2000) was developed by EIC and will be used with Galactic Industries Grams/32 version 5 software for spectral processing. The RS2000 and Grams/32 are linked via dynamic data exchange

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(DDE). A Process Instruments, Inc. (Salt Lake City, UT) 670 nm laser will be used for Raman excitation. Both laser and spectrograph will be fiber optically coupled to the Raman probe via the 90-foot length of optical fiber cable. The two optical fibers are a 90 μm core silica fiber for transmitting the laser to the sample chamber and a 200 μm core silica fiber for transmitting the collected Raman signal.

OAK RIDGE NATIONAL LABORATORY

Overview

Through the TFA, the development of an EN corrosion monitoring system was initiated for ORNL. ORNL personnel in conjunction with the Hanford EN consultant (HiLine) developed and implemented a program from laboratory proof-of-principal experiments to deployment in less than two years. Late in 1999, initial proof-of-principle requirements for laboratory testing were identified to confirm the technical basis for using EN-based systems to monitor corrosion in nuclear waste tanks for ORR. Functions and requirements for deployment of a probe in a specific tank, Tank W-23, were also identified. The probe was installed into Tank W-23 during mid-2001.

The ORR waste tanks differ significantly from those at Hanford. Tank W-23 is a 50,000-gallon waste tank fabricated from welded 304L stainless-steel plates. The tank measures approximately 3.66 m (12 feet) in diameter and 18.59 m (61 feet) in length. It is housed in a concrete vault lined with stainless steel in the Bethel Valley Evaporator Storage Tank (BVEST) facility near the center of the ORNL complex (30). The BVEST facility is used to collect liquid low-level (LLLW) waste from waste stream producers at ORNL. The LLLW is reduced in volume through processing at an evaporator prior to being moved to the BVEST facility. Tank W-23 is primarily used as a storage tank for LLLW but can also serve as a feed tank for the evaporator. Because of its role as a storage tank that could see waste streams of varying composition, Tank W-23 was an ideal tank for the application of on-line corrosion monitoring. In particular, there was interest expressed in the potential corrosion of welded areas of the tank.

Laboratory Testing Program

Prior to the fabrication and deployment of the Tank W-23 system, a testing program was developed to generate baseline data that could be used to assist in the analysis of data collected from the actual in-tank system following its deployment. Testing focused on the behavior of 304L in normal tank waste solutions.

A variety of 304L and welded 304L electrode geometries were exposed to several simulated waste and pit-inducing solutions. (Table I shows a summary of tests performed.) All tests were performed at a constant temperature of 22°C. In all tests, EN data were collected from three-electrode channels at a rate of one simultaneous measurement of current and potential per second. Electrodes were electrically isolated from electrode holders with peroxide-cured EPDM O-ring type gaskets and glass-lined feed-throughs similar to those that will be used on an actual in-tank probe. The field instrumentation was used to collect, store, and process data. Short lengths of cable were used for each channel to collect the analog corrosion signal from the test cell and transmit it to the corrosion monitoring instrumentation. Approximately 60-foot cables were used in the field. Since the field system demands intrinsic safety barriers on all tank-penetrating conductors, laboratory testing also used shunt-diode type intrinsic safety barriers. The test results produced EN data for uniform corrosion and pitting corrosion that validated results from tests previously performed by others. SCC EN data were recorded in the ORNL corrosion probe development program.

Tank W-23 System

The ORNL Tank W-23 corrosion monitoring system is formed from two basic parts: the in-tank instrument tree and the ex-tank instrumentation. The in-tank instrument tree is used to deploy corrosion monitoring electrodes into the

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Table I: Summary of EN tests performed at ORNL

Test	Test Duration	Test Solution	Electrode Geometry	Electrode Surface Area
1	10/17/00 -- 10/24/00	Simulated W-23 Waste	1" Diameter 304L Bullet Shaped Electrodes	~20 cm ²
2	10/17/00 -- 10/24/00	Simulated W-23 Waste	1" Diameter 304L C-rings	~47 cm ²
3	10/17/00 -- 10/24/00	Simulated W-23 Waste	1" x 2" 304L Flat Coupons	~31 cm ²
4	10/17/00 -- 10/24/00	Simulated W-23 Waste	1" x 2" 304L Flat Coupons with Autogenous Weld	~31 cm ²
5	10/24/00 -- 10/26/00	100 mg/L FeCl ₃	1" x 2" 304L Flat Coupons	~31 cm ²
6	10/24/00 -- 10/26/00	10 mg/L FeCl ₃	1" x 2" 304L Flat Coupons	~31 cm ²
7	10/24/00 -- 10/26/00	1 mg/L FeCl ₃	1" x 2" 304L Flat Coupons	~31 cm ²
8	10/24/00 -- 10/26/00	0.1 mg/L FeCl ₃	1" x 2" 304L Flat Coupons	~31 cm ²
9	10/26/00 -- 10/29/00	Simulated W-23 Waste	1" Diameter 304L Bullet Shaped Electrodes	~20 cm ²
10	10/26/00 -- 10/29/00	Simulated W-23 Waste	1" Diameter 304L C-rings	~47 cm ²
11	10/26/00 -- 11/02/00	100 g/L NaCl	1" x 2" 304L Flat Coupons with Autogenous Weld	~31 cm ²
12	10/26/00 -- 11/02/00	10 g/L NaCl	1" x 2" 304L Flat Coupons with Autogenous Weld	~31 cm ²
13	10/29/00 -- 11/02/00	Simulated W-23 Waste	1" Diameter 304L Bullet Shaped Electrodes with 308L Weld Band	~20 cm ²
14	10/29/00 -- 11/02/00	Simulated W-23 Waste	1" Diameter 304L Bullet Shaped Electrodes with 308L Weld Band	~20 cm ²

tank. General in-tank design features include, but are not limited to, the following: the probe is fabricated from 304/304L dual rated stainless steel; the probe is designed to fit through a 10.2-cm (4.0 in.) diameter riser; the probe design facilitates decontamination by minimizing areas of liquid retention; all materials are capable of withstanding temperature ranges up to 100°C; all materials are capable of withstanding liquid phase pH ranges from 7 to 14; all materials are capable of withstanding radiation levels up to 1000 R/hr; conductor/feed-through connections are angled to withstand probe flexure; system collects SCC EN data from two channels of 304L C-ring type electrodes; one electrode of each C-ring array is pre-cracked and strained prior to immersion; system collects pitting EN data from two channels of 304L "bullet" type electrodes; bullet electrodes are fabricated from two pieces of 304L bar stock welded with 308L filler prior to final machining; EPDM O-ring gaskets are used to electrically isolate electrodes from probe body; and, the probe body serves as grounded shield to reduce unwanted interference in the data.

The following are the primary design features of the ex-tank instrumentation software/hardware used to collect data from the in-tank instrument tree: current and voltage EN data are collected in an automated, user configurable fashion; system simultaneously monitors four channels of EN electrodes (not multiplexed); system is capable of periodically conducting LPR scans at user-configurable intervals; data collection hardware is housed in a custom enclosure placed in a climate controlled instrument building adjacent to BVEST W-23; data are stored locally on data collection computer for analysis; above ground wiring uses driven shields to reduce external electrostatic noise pickup; shunt-diode type intrinsic safety barriers are used on all tank-penetrating conductors; the operating software is compatible with site standard desktop PCs; and all data is stored in ODBC and SQL compatible databases.

System Operation

Since its installation, the Tank W-23 EN system has collected over 14 million points of data on the four channels being monitored. Table II shows the configuration of each of the eight channels on the in-tank probe.

In general, the Tank W-23 corrosion monitoring system is behaving as expected and collecting data on a full-time basis with no obvious external electrical interference or data corruption. Upon installation of the in-tank probe, all channels behaved as predicted. Channels one and two did not show a great disturbance in corrosion behavior since the electrodes did not enter the waste. Channels three and four reacted to their introduction to the waste with a large

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transient in current and voltage and followed by a slow passivation process that started equilibrating after five weeks of operation.

Table II: Tank W-23 corrosion probe electrode layout and descriptions

Channel	Distance from Top of Riser	Electrode Type	Environment	Can Detect
1	79.5"	Bullet	Tank Vapor Space	Pitting
2	118.25"	C-ring	Tank Vapor Space	Pitting and Stress Corrosion Cracking
3	157"	Bullet	Supernate	Pitting
4	195.75"	C-ring	Supernate	Pitting and Stress Corrosion Cracking

Field data collected on channels one and two through the first five weeks of operation indicate periods of corrosion when atmospheric humidity is sufficient to coat the electrodes with enough electrolyte to allow the conduction of current. These time periods are intermittent in nature, but become more consistent over time as the electrode surfaces form thicker passive layers which facilitate the retention of moisture. However, no laboratory data on electrodes immersed in this vapor space environment or a simulated vapor space environment has been collected to verify this hypothesis.

Field data collected on channels three and four through the first five weeks of operation show EN data indicative of low rates of uniform corrosion. This result is consistent with tank design considerations (304L selected as tank material for corrosion resistance) and with data collected on 304L electrodes immersed in simulated Tank W-23 waste. Field data does contain the occasional transient that is thought to be indicative of pit initiation. This result is consistent with laboratory data done in support of the development of the field system. The occasional pit initiation transients in the field data are similar to the transients from the lab data. These type of transients did not result in any gross damage to the surface of the electrodes used in laboratory waste simulant testing and are therefore unlikely to be indicative of the onset of corrosion damage in the tank. No pit growth transients have been recorded in the field data. The EN transients that stand out from the background are not necessarily associated with pit initiation or other corrosion phenomena. Other surface disturbances such as gas bubble formation and release can create transients in EN data.

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Overview

Starting in 2001, the TFA provided funding to AEA Technologies to perform initial studies using an EN-based corrosion monitoring system in simulated INEEL tank waste. This waste differs from all other DOE high-level waste. Engineers from SRS, Hanford and HiLine have provided INEEL with technical support in defining the testing program and equipment selection.

Spent nuclear fuel (SNF) was reprocessed at the INEEL from 1953 through 1992. This reprocessing produced mixed liquid waste, which was stored in the Idaho Nuclear Technology and Engineering Center (INTEC) tank farm. This tank farm consists of eleven, 300,000-gallon stainless steel tanks placed within underground concrete vaults. The waste tanks were constructed from 1951 to 1964 and were put into radioactive liquid waste service from 1953 to 1966. The first two tanks (WM-180 and -181) were constructed of 347 stainless steel with the remaining tanks (WM-182 through WM-190) constructed out of 304L stainless steel. From 1963 through 2000, most of this liquid waste was removed from the tank farm and solidified through a calcination process.

Two types of liquid waste have been stored, high-level waste and sodium bearing waste (SBW). The composition of the high-level waste was dependent on the SNF type being processed (aluminum, stainless steel or zircalloy cladding). The SBW resulted from processing and decontamination activities that made extensive use of sodium-based chemicals such as sodium hydroxide and sodium carbonate. Approximately 960,000 gallons of predominately SBW wastes are still stored in the INTEC tanks. The INTEC wastes have been generally characterized by large

concentrations of nitrates and dissolved metals such as aluminum, potassium, and sodium with small concentrations of sulfates, chlorides, chromium and nickel. A typical SBW composition is shown in Table III (31).

Table III. Typical Sodium Bearing Waste Chemistry

Major Species	Molarity	Major Species	Molarity	Major Species	Molarity
Acid (H ⁺)	1.28	Potassium (K)	0.17	Nitrate (NO ₃)	4.50
Aluminum (Al)	0.57	Iron (Fe)	0.002	Sulfate (SO ₄)	0.043
Boron (B)	0.017	Mercury (Hg)	0.0013	Uranium (U)	66.4 (mg/L)
Cadmium (Cd)	0.001	Fluoride (F)	0.04	Undissolved Solids	2.4 (g/L)
Sodium (Na)	1.50	Chloride (Cl)	1000 (mg/L)	Liquid Density	1.25 g/mL

Small amounts of fission products and transuranic elements are also in the waste. The Cl⁻ and F⁻ ions can cause localized corrosion and stress corrosion cracking of stainless steel alloys. The tanks also contain solids found in a sludge layer on the tank bottom and adhere to the tank walls in some of the tanks.

The corrosion-monitoring program for these tanks has consisted of remote visual inspections using video cameras and retrieval of corrosion coupons for determining corrosion rates and localized corrosion tendencies. The historical corrosion performance of the tanks through 1988 has shown very low rates of general corrosion (5.3×10^{-2} mils/year maximum) with no evidence of localized corrosion such as pitting or stress corrosion cracking (32-35). However, recent inspections of corrosion coupons in tanks WM-183 and WM-187 revealed initiation of pitting (36,37). Tank WM-183 is being taken out of service but WM-187 (along with tanks WM-180,-181,-188 and -189) will see continued service for an estimated twenty years. It should be noted that the two oldest tanks (WM-180 and -181) would continue in service.

Background - AEA Technology EN Work

A program was recently initiated to ascertain the applicability of EN-based systems to stainless steel tanks containing acidic waste solutions containing high concentrations of chlorides and fluorides. AEA Technologies conducted the laboratory research program which included procurement and setup of Concerto equipment. The system is similar to those installed at the other DOE sites. The work involved initial baseline data of 304L stainless steel in nitric acid chemistry (Table IV), and testing of a simulated INEEL SBW waste (Table V)(38).

Table IV. Initial Baseline Testing

Test No.	Solution	Temperature	Time	Electrode	Surface	Coupons
1A	Nitric Acid	25 °C	5 days	Cylindrical pins (5 cm ²)	Smooth (1200 grit)	None
1B	Nitric Acid	50 °C	5 days	Cylindrical pins (5 cm ²)	Smooth (1200 grit)	None

Table V. Baseline Testing of Simulant Solutions

Test No.	Solution	Temperature	Time	Electrode	Surface	Coupons
2A	Simulant solution	25 °C	10 days	Cylindrical pins (5 cm ²)	Smooth (1200 grit)	After 2, 5, 10 days
2B	Simulant solution	50 °C	10 days	Cylindrical pins (5 cm ²)	Smooth (1200 grit)	After 2, 5, 10 days

Table VI describes additional tests that evaluated EN response to sample surface finish and stress state.

Table VI. Simulant Testing, Effect of Sample Configuration

Test No.	Solution	Temperature	Time	Electrode	Surface	Coupons
3A	Simulant solution	25 °C	2 days	Cylindrical pins (5 cm ²)	Rough (120 grit)	After 2 days
3B	Simulant solution	50 °C	2 days	Cylindrical pins (5 cm ²)	Rough (120 grit)	After 2 days
3C	Simulant solution	25 °C	5 days	Open tube (20 mm OD)	Smooth (1200 grit)	After 5 days
3D	Simulant solution	50 °C	5 days	Open tube (20 mm OD)	Smooth (1200 grit)	After 5 days
3E	Simulant solution	25 °C	5 days	Stressed C (20 mm OD)	Smooth (1200 grit)	After 5 days
3F	Simulant solution	50 °C	5 days	Stressed C (20 mm OD)	Smooth (1200 grit)	After 5 days

Research Results

Phase 1 (1A, 1B) -- The response of 304L stainless steel to nitric acid at 25 and 50° was expected to show passivation of the metal surface with very low rates of general corrosion. The current plot at 25° C is stable and featureless. The current plot at 50° exhibited more fluctuations. SEM examination of the test electrodes showed possible pit initiation at 25°C and some pit initiation at 50°C.

Phase 2 (2A, 2B) -- The EN results were difficult to interpret due to difficulties with data analysis of the larger files. The SEM micrographs of the test electrodes at 25 and 50° exhibited some pit and crevice corrosion initiation. The removable corrosion coupons showed no evidence of localized corrosion with very few surface features evident.

Phase 3 (3A, 3B) -- The system had not fully equilibrated at the end of the two-day test. The EN plots at the end of the test show rapid, microscopic variation in the current signal. This could be related to localized corrosion initiation events. The SEM micrographs of the electrodes at 25 and 50°C show pit initiation and crevice corrosion in the rough surface. The removable corrosion coupons showed no visible corrosion.

Phase 3 (3C, 3D, 3E and 3F) -- The statistical evaluation of the data shows that these systems had just come to equilibrium at the end of the test period. The EN plots at the end of the test again showed rapid, microscopic variation in the current signal. One stressed C-ring electrode (3C at 25°) showed evidence of crack initiation indicative of stress corrosion cracking near the bottom of the v-notch. The removable corrosion coupons showed no visible corrosion.

SUMMARY

The Hanford corrosion monitoring system design has evolved steadily since the prototype probe was installed in 1996. An effort has been made with each successive design to correct problems detected with previously installed systems. Additionally, each successive system has made better use of tank riser space by incorporating other pieces of equipment on the probe body to compliment the existing corrosion monitoring components.

Currently, data are being collected from the four systems installed in the 241-AN tank farm at Hanford. Data are collected full-time, on a once-every-other-second basis on each channel of each system (one simultaneous measurement of current noise and voltage noise every other second on each channel on each system). Data is logged locally on the computer associated with each in-tank probe. Data are checked at least once per day to verify system operation. Troubleshooting and system resets are performed as necessary. Each week, data is downloaded from each system and processed using the operating software. Periodically, findings of these analyses are reported to management. Currently, all data downloading, processing, analysis and reporting is handled by the engineering organization or its subcontractors.

In 2001, the Hanford EN-based corrosion monitoring program was reviewed by a panel of experts from academia and industry to provide a path forward for the site for corrosion monitoring and integrity management of the tanks.

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The review panel found that further basic development is still necessary, particularly in the areas of data interpretation, sensor refinement and equipment reliability. Additionally, the site needs to generate a clear vision and integration plan for the data produced by the corrosion monitoring systems prior to turnover to operations.

Through the Tanks Focus Area the lessons learned from the development of the Hanford EN probe systems have been applied to the development of probes for SRS and ORNL, as well as preliminary research for INEEL applications. Although site deployment conditions dictate specific design differences the basic system considerations have been incorporated into systems for the other sites.

The combined probe for SRS underwent a thorough cold demonstration during 2001 and installation on Tank 43 is planned for mid-2002. The deployment platform will be mounted on Tank 43-H with the ex-tank systems installed in a mobile trailer located in close proximity to the tank top.

The system for ORR was deployed in BVEST tank W-23 on June 21, 2001 and is operating successfully. Prior to the fabrication and deployment of the Tank W-23 system, laboratory testing was performed to characterize the behavior of 304L stainless steel in simulated Tank W-23 waste solutions. This testing was performed primarily to support field data interpretation. Testing at ORNL produced EN data for uniform corrosion and pitting corrosion that validated results from tests previously performed by others. In general, the Tank W-23 corrosion monitoring system is behaving as expected and collecting data on a full-time basis with no obvious external electrical interference or data corruption.

At INEEL, the results from the preliminary test program show the 304L stainless steel alloys passivate and show low currents and no signal features in nitric acid exposure. The EN signal features increase with exposure to simulated SBW and the temperature increase from 25 to 50°C. There were anomalies in pitting and crevice initiation tendencies between the electrodes and the removable samples. The initial testing of the stressed C-ring electrodes was not able to identify a characteristic signature for SCC. The un-stressed C-ring experiments indicated a gradual evolution of the electrochemical potential and current. SEM microscopy identified one stressed C-ring electrode that exhibited SCC behavior as compared to passive behavior in the other stressed and unstressed C-ring electrodes. Recommendations for additional work include: 1) Potentiodynamic testing of the alloys used for electrodes and coupons to establish baseline localized corrosion performance 2) Perform testing in known, aggressive environments where pitting and SCC would occur to establish baseline EN signatures 3) Perform additional work on data handling of large data sets, storage, export, and numerical processing,

The AEA Technologies testing has demonstrated that EN measurements can be made with the austenitic stainless steels used in the INEEL HLW tank construction when exposed to simulated waste solutions. The program will continue with additional testing at AEA Technology and the INEEL. The program goal is to design and fabricate an EN system for deployment in an INEEL HLW tank.

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